The Effect of Hydrogen Annealing on the Impurity Content of Alumina-Forming Alloys

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Abstract.

Previously, the effect of hydrogen annealing on increasing the adhesion of Al_2O_3 scales had been related to the effective desulfurization that occured during this process. The simultaneous reduction of other impurities has now been re-examined for up to 20 impurity elements in the case of five different alloys (NiCrAl, FeCrAl, PWA 1480, Rene'142, and Rene'N5). Hydrogen annealing produced measurable reductions in elemental concentration for B, C, Na, Mg, P, K, Sr, or Sn in varying degrees for at least one and up to three of these alloys. No single element was reduced by hydrogen annealing for all the alloys except sulfur. In many cases spalling occurred at low levels of these other impurities, while in other cases the scales were adherent at high levels of the impurities. No impurity besides sulfur was strongly correlated with adhesion.

Introduction.

Early theories of Al_2O_3 scale adhesion most commonly invoked mechanisms based on mechanical keying (pegging)[1], decreased wrinkling (growth stress)[2], and decreased interfacial voids (vacancy sink).[3] Thus much attention had been given to reactive element effects on the morphology and transport within the scales and the marked changes that took place with the addition of reactive elements. A consensus was elusive because of various exceptions and the inability to control a single phenomenon during doping without also incurring at least one other simultaneous manifestation of the reactive element effect.

Some time ago the reactive element effect on Al_2O_3 scale adhesion to NiCrAl alloys was proposed to result from the prevention of sulfur segregation at the oxide-metal interface.[4] In proof of this mechanism, a number of studies have documented a substantial improvement in scale adhesion by reducing the sulfur content of undoped alloys. One experiment used repeated oxidation and polishing to reduce the sulfur content from about 10 to below 3 ppm of sulfur.[5] Another used ultra high purity materials in the casting to produce an alloy with about 2 ppm sulfur.[6] The most effective technique employed hydrogen annealing to reach sulfur levels of 0.1 ppm or below in many instances.[7-12] While it can be directly confirmed that these techniques produced scale adhesion without reactive elements, the role of other impurities was not directly addressed.

Recently it has been suggested that hydrogen annealing removes not only sulfur but also all other impurities. It can thus lead to adhesion by preventing deleterious morphological effects caused by Ca or Na impurities.[13] In support of this proposal, NaOH and $Ca(OH)_2$ surface deposits were shown to dramatically increase the amount of Al_2O_3 scale wrinkling and detachment on FeCrAl and FeCrAlY alloys formed at 1100°C. In unmodified alloys, with nominal levels of Ca and Na, the impurity effects are said to be neutralized by the presence of Y.[13]

The purpose of the present study is to review the impurity levels of alloys before and after hydrogen annealing in order to assess any strong correlation with an adhesion effect. In many cases a reasonably large sampling of impurity elements was obtained at the same time of the sulfur analyses, but had not been reported in the original studies. This paper tabulates the chemical analyses of those trace impurities, when available.

Experimental.

Test coupons on the order of 1-2 mm thick, 10 mm wide and 10-20 mm long were machined from arc melted buttons of Ni-15Cr-13Al and Fe-18Cr-6Al, polycrystalline stock of PWA 1480, directionally solidified Rene'142, and single crystal Rene'N5 (+Y) superalloys. Hydrogen annealing was performed in flowing 5%H_2/Ar in a leak-tight alumina tube furnace for NiCrAl, FeCrAl, and PWA 1480. Rene'142 and Rene'N5 were annealed in flowing 100% H_2. Temperatures ranged from 1000-1300°C and times ranged from 8-100 hr. Cyclic oxidation was performed at 1100° or 1150°C with 1 hr. heating cycles. More experimental specifics can be obtained from the original sources.[7-12]

The chemical analyses of impurities were obtained by glow discharge mass spectroscopy (GDMS, Shiva Technologies). At low levels (1 ppm) this technique is typically quoted at accuracy levels of ± 25%, yet it is extremely sensitive to absolute values at the ppb level. In the special case of sulfur analyses, a recent isotope dilution-thermal ionization mass spectroscopy study (ID-TIMS) measured sulfur levels in a melt desulfurized PWA 1484 alloy to be 0.5 ± 0.2 (2σ) ppmw (Dr. W. Robert Kelly, National Institute of Science and Technology) compared to 0.12 ± 0.02 (1σ) determined by GDMS. The trends established in this paper deal with impurity reductions by over 50% of the initial amounts, for impurities initially present at levels ≥1 ppmw.

As will be evident in the discussion, two analysis schemes are reported for the various alloys. In the full scan scheme, results were obtained for 74 elements. Values obtained for the primary constituents and other transition metal impurities did not change appreciably and are not reported here. Emphasis is placed on alkali and alkaline earth elements, semi-metals, heavy metals, chalcogens, and halogens. In the partial scan scheme, only eight impurity elements were analyzed other than the primary constituents and transition metals.

Results.

Tables I-III summarize the significant impurity levels measured for the alloys studied. These impurities are termed 'active' since they were altered in at least one case. The hydrogen annealing conditions and subsequent cyclic oxidation testing and weight change are included in the column headings. In general, a substantial improvement in cyclic oxidation occurs with hydrogen annealing, except as specifically noted later. The chemical data entries have been boldfaced for elements present at 1 ppm or greater, where a significant reduction has taken place due to hydrogen annealing. There are five instances where the impurity level actually 'increased' with annealing, suggesting random fluctuations in low levels of impurities.

The effect of hydrogen annealing on the impurity content of NiCrAl and FeCrAl alloys is summarized in Table I. For NiCrAl: P was reduced the most, Mg and Sn were reduced measurably, B was reduced slightly, and Na, K, Ca, and Sr not at all. Sulfur was reduced from 7.5 to 0.02 ppm. Cyclic oxidation showed a substantial improvement with hydrogen annealing, but was far from being ideal.

Conversely, for FeCrAl: Na, K, Ca, and Sr were now reduced measurably, while there was no change for B, Mg, P, or Sn. Sulfur was reduced from 12 to 0.1 ppm or less. Cyclic oxidation revealed a remarkable, long term, 1700 hr. beneficial effect that was essentially as effective as Y additions.[11]

(Other elements not shown on this table were also analyzed: For both NiCrAl and FeCrAl samples, Cl was less than 2 ppm and was unchanged by hydrogen annealing. Sb, Ba, and Hg were < 0.1 ppm and were unchanged. Li, Be, F, Tl, Bi, Pb, Rb, and Cs were all < 0.01 ppm and were also unchanged by hydrogen annealing).

The effect of hydrogen annealing on the impurity contents of three superalloys is shown in Table II. For PWA 1480, Mg was reduced somewhat. There was essentially no change for B, Na, Si, P, K, Ca, Ga, or Sr. Sulfur was reduced from 7.5 to less than 0.1 ppm and excellent cyclic oxidation resistance ensued.

In Rene'142, B and C were reduced substantially from very high intentionally doped levels. Mg was also reduced measurably, but Si, P, K, Ca, Ga, and Sr were not. Sulfur was reduced from 6 to 0.3 ppm. Accordingly, the cyclic oxidation resistance was markedly improved in this relatively severe 1150°C, 1000 hr. test.[10]

The B and C impurities were reduced remarkably for Rene'N5 as well. Na, Mg, Si, P, K, Ca, Ga, and Sr were not measurably reduced or were at low levels initially. Sulfur remained at the 3-4 ppm level because the samples shown were also doped with Y. Accordingly, no change in cyclic oxidation resistance was observed because the samples were adherent initially.[10]

Table III summarizes a subset of chemistry scans from of a partial data set of 22 PWA 1480 samples tested in a hydrogen annealing matrix. This data set has been arranged in order of increasing weight change to indicate improving oxidation resistance. Mg was reduced slightly, and Si, P, K, Ga, In, and Sn remained unchanged. Basically the same results were obtained for the other 16 samples. Sulfur was progressively reduced from about 6 to below 0.5 ppm in many instances. The cyclic oxidation resistance also progressively improved, and all seven samples from the full data set with less than 0.3 ppm sulfur showed excellent cyclic oxidation resistance.[12]

A survey of the above tables identifies the impurities removed by hydrogen annealing. These are now considered as candidates for controlling scale adhesion. The compositional changes are summarized in Table IV, where a ✓ indicates a reduction of at least 1 ppm, an X indicates virtually no change, and n.a. = not analyzed. Other elements analyzed, but not listed, never showed a significant reduction due to hydrogen annealing.

Looking across the table rows, it can be seen that for each alloy at least one element in addition to sulfur was reduced by hydrogen annealing. Alternatively, the predominance of one single impurity can be determined by looking down the columns. At a glance it can be seen that B was reduced by hydrogen annealing for three alloys, but not for two others; Na, Ca, and Sr for one alloy, but not for four others; Mg for three alloys, but not for three others; P and K for one alloy each, but not for five others; and Sn for one alloy, but not for two others.

At the bottom of Table IV, a ✓ indicates which element was decreased by hydrogen annealing in **all** the alloys in which it was measured. C was reduced in the two superalloys for which it was measured. But C removal by hydrogen annealing was not prevented by the presence of Y (e.g., for N5 in Table II). Thus C appears to be mobile even in the presence of Y and changes in C content are not likely to provide a strong correlation with adhesion. The other elements remained unchanged by hydrogen annealing (X) in at least two other cases.

This is also shown numerically in Table V where the impurity levels before and after hydrogen annealing have been summarized. Boldface is used to indicate where significant reductions have occurred. In an attempt to resolve a critical level needed for spallation in all the alloys, the lowest as-received impurity level of a given element is given near the bottom of the chart. For seven of these ten impurities (B, Na, Mg, K, Ca, Sr, Sn), this amount is ≤ 1 ppm, indicating that it is not likely that these elements were universally responsible for poor scale adhesion. Conversely, to determine the upper limit tolerable for adhesion, the highest amount of an impurity after hydrogen annealing is also shown at the bottom of the chart. For C and P, a relatively high amount of impurity (> 50 ppm) sometimes existed after hydrogen annealing, indicating that these elements were probably not controlling adhesion as well. Finally, the maximum level from the five alloys after annealing is higher for most elements than the minimum from the as-received materials. This is counter to the trend needed to establish other impurity effects on adhesion due to removal by hydrogen annealing.

While these tables do not conclusively exclude the potential effects of other impurity elements on scale adhesion, they do show only sporadic reductions with hydrogen annealing. At the very least, there was no one element that was consistently removed by hydrogen annealing and thereby associated with improved adhesion. In order to construct an alternative explanation to the sulfur effect, different impurity elements would have to be invoked for different alloys or specimens. Changing the mechanism each time to fit the alloy or individual test is cumbersome at best.

Except of course for sulfur. Sulfur was reduced in all the alloys. Oxide adhesion was dramatically improved only when the sulfur content had been reduced to below 1 ppm. In fact, the adhesion behavior was often equivalent to that obtained by adding Y.

Furthermore, as shown in Table III, the **degree** of adhesion follows the **degree** of desulfurization. This trend was previously documented in more detail for 22 samples of PWA 1480 with varying sulfur contents.[12] Here an oxide adhesion map for PWA 1480 was constructed that shows the strong direct relationship between the amount of sulfur available for segregation and cyclic oxidation weight loss behavior.

Discussion.

The effect of ppm levels of impurities on bulk material properties can be substantial when there is also a strong tendency of the impurity to segregate, e.g., at interfaces or grain boundaries. Of the impurities mentioned above, S, B, C, P, In, and Sn are known segregant elements in Fe and Ni alloys, whereas intentionally doped bulk Al_2O_3 most often exhibits grain boundary segregation of the dopant elements (e.g., Mg, Ca, Sc, Ti, Y, La, Si). Mg is difficult to measure experimentally because of its low sensitivity in electron spectroscopies, whereas Ca and Si have long been recognized as impurity segregants in bulk Al_2O_3.[14].

In oxidation studies, P had been found once to segregate at the Al_2O_3-NiCrAl interface in an early study.[15] More recently, C has been found to segregate at the Al_2O_3-FeCrAl(Y) interface[16-18], but almost equally under adherent or non-adherent scales formed on as-received, hydrogen annealed, or Y-doped alloys. In an extensive study of FeCrAl oxidation, the cation grain boundary segregation in the scales was measured for intentional dopants of 2000 ppm (atomic) of many reactive or transition metal elements.[19] This study never found any of the cation impurities mentioned in Tables I to III to segregate in the scales, except when the alloy was heavily doped with Ca at 2000 ppm. Although Mg segregation was not observed when doped at 2000 ppm, it was observed to migrate to the gas surface and improved adhesion somewhat.

In summary, there is little direct evidence that foreign impurity elements are measurably concentrated in the scales or at the interface, unless they are intentionally added to the alloy in comparatively large quantities. There appears to be no single cation impurity widely associated with control of wrinkling, growth stress, or adhesion in the scales. Conversely, there is general agreement and corroboration that the effective reactive element dopants do segregate at the oxide-metal interface and scale grain boundaries, but, if anything, this segregation is beneficial (e.g., Pint).[19-21] It has been long been proposed that reactive element segregation is responsible for typical reactive element effects on scale morphology, transport mechanisms, and scale growth.[2, 22, 23, 19]

According to the alternative impurity explanation, the reactive elements should also have a strong affinity for the foreign cations,[13] thereby preventing them from degrading the scale. Such an interaction would be needed if cation impurity control is the mechanism by which reactive elements produce adhesion. Thus, for example, Y reactivity with B, Na, Mg, P, K, Ca, Sr, or Sn should be relatively high, as is the Y-S interaction.

But in support of the existing impurity mechanism, sulfur has repeatedly been found to segregate strongly at free surfaces and intact oxide-metal interfaces, with an enrichment ratio on the order of 10,000.[18] This segregation has largely been curtailed when reactive elements are present or when the sulfur level has been reduced to ≤ 0.1 ppm by hydrogen annealing.[17, 24] Thus a complete (multiple) correlation exists between hydrogen annealing, bulk sulfur content, sulfur segregation levels, and scale adhesion in oxidation tests. The same is not true for any of the other impurities.

An additional concern with the foreign cation proposal is the **necessity** of growth stress, i.e., wrinkling, to cause decohesion. The difficulty is that the high (4-6 GPa) residual stresses measured for non-adherent scales formed on undoped alloys is no higher than those in adherent alumina scales formed on doped alloys.[25] High growth stresses of about 1 GPa were indeed resolved in adherent scales formed on a FeCrAlY alloy.[26] Thus growth stress does not seem to be the primary player in causing spallation, although it certainly contributes to thermal stresses that eventually damage all scales, whether they spall at the interface or remain adherent and develop wedge cracks.

It is generally true that flatter scales are associated with adherence, especially in reference to the planar scales formed on alloys doped with reactive elements and also those formed on desulfurized alloys. But it is also true that relatively flat scales have occasionally been observed to spall off undoped alloys (without interfacial voidage). This occurred for thinner scales formed on NiCrAl after 1 hr at 1100°C, before extensive buckling had taken place[7] or for a scale formed on an electropolished FeCrAl surface after 24 hr. at 1200°C.[27] Conversely, some systems may exhibit extremely convoluted scales and still remain quite adherent (FeCrAlTi and hydrogen annealed FeCrAl, after 1700 1-hr cycles at 1100°C[11] or CoCrAlY deformed at a strain rate of 0.63% per hr at 1100°C for 16 hr).[28]

This is not to say that the dramatic buckling and void formation commonly observed is not an important contribution to the failure of that scale, but that they are not necessary for interfacial spalling, whereas sulfur segregation is. There is no apparent mechanism available suggesting how indigenous sulfur impurities might affect morphology, transport, or growth stress in the scales, if it does at all, except perhaps by allowing delamination at temperature, with subsequent plastic deformation of the scale.

Concluding Remarks.

While it may be hardly possible to desulfurize an alloy by hydrogen annealing without also reducing some other impurity, the preceding data show that there is no other impurity that appears to be generally present for non-adherent scales and generally absent for adherent scales. There is no consistent removal of such foreign elements by hydrogen annealing except for sulfur. AES and AEM studies have not identified the common presence of any other impurity in the scale or at the interface of undoped alloys exhibiting typical interfacial spallation.

The strength of the argument based on the sulfur mechanism stems from an integrated consistency of segregation, interfacial adhesion, and reactive element thermodynamic ‘gettering’ aspects, even though the exact atomistics or quantum theories of bond strength may not be fully developed. It is further strengthened by critical experiments where adhesion was produced by reducing the sulfur content, without reactive elements. There are no experiments known to this author where alumina scale adhesion, comparable to that conferred by reactive element doping, was produced by any other means.

In order to meaningfully search for and conclusively prove alternative explanations to the reactive element effect (i.e., the sulfur effect), adhesion should be demonstrated as a direct result of the new mechanism, without adding reactive elements and without desulfurization. For example, a potential critical experiment would be to produce very adherent scales in an undoped alloy with a strictly controlled sulfur level, fixed at about 5-10 ppm, simply by removing the native Na, Ca, etc. impurities.

For engineering applications of NiCrAl, CoCrAl, and FeCrAl alloys, the role of sulfur is primarily of scientific interest since these alloys can generally be doped with reactive elements quite effectively with little expense or difficulty. The same is not true for single crystal superalloys, which can be made to be quite oxidation resistant if the inner Al_2O_3 scale layer is adherent. Here Y has been found to be the most effective dopant, but may cause considerable difficulty in processing and product yield because of its high reactivity with mold materials. As an alternative solution, melt desulfurization processes using Ca-rich materials currently produce commercially available low sulfur (0.5 ppmw) superalloys. These have been shown to have excellent cyclic oxidation resistance at 1100°-1150°C for thousands of hours and thus provide an important practical application of understanding adhesion mechanisms.

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Table I. The effect of hydrogen annealing on active impurity content and cyclic oxidation for NiCrAl and FeCrAl alloys. (GDMS analyses in ppmw; boldface indicates significant reductions).

alloy	NiCrAl	NiCrAl		FeCrAl	FeCrAl-1	FeCrAl-2
H_2-anneal, °C/hr	as-cast	1200/100		as-rec'd	1200/100	1200/100
oxidation,°C/hr	1100/200	1100/200		1100/225	1100/1700	1100/1700
ΔW/A, mg/cm^2	-21.91	-2.71		4.7	1.2	1.2
S	**7.5**	**0.02**		**12.5**	**0.1**	**0.05**
B	**1.5**	**0.5**		0.2	0.3	0.2
Na	0.15	1.5		**6**	**0.4**	**0.8**
Mg	**3**	**0.02**		0.2	0.02	0.08
Si	210	170		380	150	230
P	**60**	**0.5**		2.5	4	5
K	0.09	0.07		**3**	**0.1**	**0.1**
Ca	0.1	0.2		**2**	**0.1**	**0.5**
Ga	30	88		23	25	20
As	0.3	0.9		0.3	0.5	0.7
Sr	<0.05	<0.05		**5**	**<0.05**	**<0.05**
Sn	5	1		1	1	0.2

Table II. The effect of hydrogen annealing on active impurity content and cyclic oxidation for three Ni-base superalloys. (GDMS analyses in ppmw; boldface indicates significant reductions).

alloy	PWA 1480				Rene'142			Rene'N5	
sample I.D.	50-01	7H	42H		5C	7		5C	7
H_2-anneal, °C/hr	as rec'd	1200/100	1200/100		as-rec'd	1280/100		as-rec'd	1280/100
oxidation,°C/hr	1100/500	1100/500	1100/500		1150/1000	1150/1000		1150/1000	1150/1000
$\Delta W/A$, mg/cm^2	**-66.63**	**0.65**	**0.75**		**-94.76**	**-2.1**		**0.61**	**0.59**
S	**7.5**	**0.01**	**0.08**		**6.1**	**0.3**		**3.2**	**3.5**
B	1.5	0.7	1.2		**180**	**0.35**		**42**	**0.005**
C	n.a.	n.a.	n.a.		**2300**	**50**		**330**	**30**
N	n.a.	n.a.	n.a.		**2.5**	**0.1**		0.5	0.2
Na	0.04	0.03	0.03		0.03	0.3		<0.01	0.05
Mg	**3.5**	**0.9**	**1.8**		**3.5**	**0.2**		0.15	0.05
Si	225	320	360		170	150		520	480
P	68	50	64		5.4	3		3.9	4.5
K	0.03	0.03	0.05		0.05	0.3		0.09	0.05
Ca	<0.1	<0.1	<0.1		<0.05	0.09		<0.05	<0.05
Ga	30	37	33		<0.1	17		23	15

Table III. The progressive effects of six hydrogen annealing conditions (out of 22 total) on active impurity content (partial scan) and cyclic oxidation for PWA1480 samples. (GDMS analyses in ppmw; boldface indicates significant reductions).

sample I.D.	20-7c1	20-1	50-2	20-5	20-3	20-9
H_2-anneal, °C/hr	as-rec'd	1000/20	1300/20	1200/8	1200/20	1200/50
oxidation,°C/hr	1100/500	1100/500	1100/500	1100/500	1100/500	1100/500
ΔW/A, mg/cm^2	-32.86	-24.99	-8.44	-2.62	0.585	0.65
S	**6.7**	**3.9**	**1.8**	**0.8**	**0.28**	**0.05**
Mg	2.2	2	0.34	1.5	1.4	0.9
Si	220	230	230	210	210	210
P	66	74	56	58	60	76
K	0.02	<.01	0.02	<.01	0.02	<.01
Ga	17	16	17	17	17	15
In	<.01	0.01	0.02	<.01	<.01	0.01
Sn	2.8	2.4	4.1	2.4	2.6	2.2

Table IV. Incidence chart for active impurity removal by hydrogen annealing for five alloys. (✓ indicates significant reductions, **X** indicates little or no reduction, n.a. = not analyzed). Bottom of chart indicates the relative occurrence of significant reductions and a ✓ if reduced for all alloys measured and an **X** if not.

	S	**B**	**C**	**Na**	**Mg**	**P**	**K**	**Ca**	**Sr**	**Sn**
NiCrAl	✓	✓	n.a.	X	✓	✓	X	X	X	✓
FeCrAl	✓	X	n.a.	✓	X	X	✓	✓	✓	X
1480 (1)	✓	X	n.a.	X	✓	X	X	X	X	n.a.
1480 (2)	✓	n.a.	n.a.	n.a.	X	X	X	n.a.	n.a.	X
Rene' 142	✓	✓	✓	X	✓	X	X	X	X	n.a.
N5 (+Y)	✓	✓	✓	X	X	X	X	X	X	n.a.
Σ	✓	**X**	✓	**X**	**X**	**X**	**X**	**X**	**X**	**X**
incidence	**6/6**	3/5	**2/2**	1/5	3/6	1/6	1/6	1/5	1/5	1/3

Table V. Summary of impurity changes due to hydrogen annealing for five alloys. Bottom of chart lists the lower limit of an element present with spallation (not annealed) and the upper limit present with adhesion (annealed). (GDMS analyses in ppmw; boldface indicates significant reductions).

	S	**B**	**C**	**Na**	**Mg**	**P**	**K**	**Ca**	**Sr**	**Sn**
NiCrAl	**7.5**	**1.5**	n.a.	0.15	**3**	**60**	0.09	0.1	<0.05	**5**
annealed	**0.02**	**0.5**	n.a.	1.5	**0.02**	**0.5**	0.07	0.2	<0.05	**1**
FeCrAl	**12.5**	0.2	n.a.	**6**	0.2	2.5	**3**	**2**	**5**	1
annealed	**0.1**	0.3	n.a.	**0.8**	0.08	5	**0.1**	**0.5**	**<0.05**	1
1480 (1)	**7.5**	1.5	n.a.	0.04	**3.5**	68	0.03	<0.1	4	n.a.
annealed	**0.08**	1.2	n.a.	0.03	**1.8**	64	0.05	<0.1	4	n.a.
1480 (2)	**6.7**	n.a.	n.a.	n.a.	2.2	66	0.02	n.a.	n.a.	2.8
annealed	**0.28**	n.a.	n.a.	n.a.	1.4	76	0.02	n.a.	n.a.	2.6
Rene' 142	**6.1**	**142**	**2300**	0.03	**3.5**	5.4	0.05	<0.05	1.5	n.a.
annealed	**0.3**	**0.35**	**50**	0.3	**0.2**	3	0.3	0.09	0.9	n.a.
N5 (+Y)	3.2	**42**	**330**	<0.01	0.15	3.9	0.09	<0.05	0.9	n.a.
annealed	3.5	**0.005**	**30**	0.05	0.05	4.5	0.05	<0.05	0.6	n.a.
limits:										
min., as-rec'd	**6.1**	0.2	**330**	<0.01	0.15	2.5	0.03	<0.05	<0.05	1
max., annealed	**0.3**	1.2	**50**	1.5	1.8	76	0.3	0.5	4	2.6